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Docket No.: 239709US23

**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

IN RE APPLICATION OF:

GROUP: 1732

Benjamin CHU, et al.

SERIAL NO: 10/674,464

EXAMINER: TENTONI

FILED: October 1, 2003

FOR: ELECTRO-BLOWING TECHNOLOGY FOR FABRICATION OF FIBROUS  
ARTICLES AND ITS APPLICATIONS OF HYALURONAN

**DECLARATION UNDER 37 C.F.R. 1.131**

COMMISSIONER FOR PATENTS  
ALEXANDRIA, VIRGINIA 22313

Sir:

Now comes Dufei Fang who deposes and states that:

1. I am a graduate of Fudan University (China) and received my Ph.D. degree in Nuclear Physics in the year 1988.
2. I have been employed by Stonybrook Technology and Applied Research since 1999 as Director of Technology.
3. I am one of the named co-inventors on the present patent application.
4. The Examiner has rejected the claims of the present patent application in light of Published US Application 2005/0067732 to Kim et al (hereafter the "Kim" reference). In order to demonstrate that we were in possession of our invention prior to the effective date of the Kim reference, I provide the following information.
5. It is my understanding that the effective prior art date of the Kim reference is November 20, 2002.
6. In August, 2002, we submitted a research proposal to the U.S. Army Small Business Innovation Research (SBIR) Program. Attached as Exhibit A is a copy of the proposal description submitted along with the Proposal Cover Sheet, dated August 14, 2002.
7. The research proposal submitted to the SBIR Program describes our invention sufficiently to show that we had conceived the invention of combining electrospinning and

melt blowing into a single process prior to the effective November 20, 2002 date of the Kim reference.

8. In particular, attention is drawn to Section 3.3 beginning at page 6 of the proposal, which describes the aspect of the proposed research which combines electrospinning and melt blowing to gain the combined effects of electrostatic repulsions and the high velocity of the gas stream on the fibers begin generated.

9. Attached also as Exhibit B is the letter from the Army dated November 11, 2002, notifying us that our proposal had been selected for negotiation and possible contract award.

10. We conceived our invention prior to the effective date of the Kim reference, as evidenced by our research proposal to the Army SBIR Program. Accordingly, the Kim reference is not available as prior art against the present application.

11. The undersigned petitioner declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of this application or any patent issuing thereon.

12. Further deponent saith not.

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2/16/2006  
Date

Small Business Innovation Research (SBIR) Program  
Proposal Cover Sheet

Proposal Number: A022-2903 Agency: Army DUNS: 101394430  
Topic Number: A02-193 CAGE:  
Proposal Title: Novel Clothing Nonwoven Liner Material - Nanofibers in Melt Blown Media

Firm:

Firm Name: Stonybrook Technology and Applied Research, Inc.  
Mail Address: P.O. Box 1336

Stony Brook, New York 11790

Website Address:

Proposed Cost: 69889 Phase: I Duration: 6  
Option Cost: 49975 Option Duration: 4

Business Certification: (Check all that apply)

Are you a small business as described in paragraph 2.2 ? YES  
Number of employees including all affiliates (average for preceding 12 months): 3  
Are you a socially or economically disadvantaged business as defined in paragraph 2.3 ? NO  
Are you a woman-owned small business as described in paragraph 2.4 ? NO  
Has this proposal been submitted to other US government agencies, or DoD components or other SBIR activity? NO  
If yes, list the name(s) of the agency, DoD component or other SBIR office and Topic Number in the space below.

Project Manager/Principal Investigator


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For any purpose other than to evaluate the proposal, this data except proposal cover sheets shall not be disclosed outside the Government and shall not be duplicated, used or disclosed in whole or in part, provided that if a contract is awarded to this proposer as a result of or in connection with the submission of this data, the Government shall have the right to duplicate, use or disclose the data to the extent provided in the funding agreement. This restriction does not limit the Government's right to use information contained in the data if it is obtained from another source without restriction. The data subject to this restriction is contained on the pages of the proposal listed on the line below.

Proprietary Information:

  
Signature of Principal Investigator

8/14/02  
Date

\_\_\_\_\_  
Signature of Corporate Business Official

\_\_\_\_\_  
Date

Technical Abstract (Limit your abstract to 200 words with no classified or proprietary information)

This Small Business Innovation Research Phase I Project aims to develop innovative key technology to combine the melt-blown process with Multi-Jet electrospinning process that can fabricate membranes with new microfiber/nanofiber hybrid morphology and can lead to a commercial scale-up process. The specific aims of this Phase I proposal are to implement several new designs to incorporate the Multi-Jet electrospinning process (patent pending) in the conventional melt-blown process. Complex and coupled processing parameters including novel spinneret assemblies, new electrode designs, and control of jet acceleration, transportation and manipulation will be considered. The unique Multi-Jet electrospinning has been developed by the PI from Stonybrook Technology and Applied Research (STAR), Inc. and scientists from the Chemistry Department in the State University of New York at Stony Brook (SUNYSB). This technology is capable of producing new nanostructured membranes with hybrid nanofiber/nanoparticle morphology, designed composition variations and 3D pattern formation.

Anticipated Benefits/Potential Commercial Applications of the Research or Development. (No classified or proprietary information)

Non-woven protective clothing with functions of non-wetting and low absorption could be used in many situations. Thus it will have high potentials for commercialization

List a maximum of 8 Key Words that describe the Project.

multi-jet Electrospinning, nanofiber, non-woven, melt-blown, co-spinning

## 1. Problems and Opportunities in Electrospinning Technology

### 1.1 Current State and Problem in Nano- and Micro-Fibers

Electrospinning is an atomization process of conducting fluid. It takes advantages of the interactions between the electrostatic field and the conducting fluid. When an external electrostatic field is applied to a conducting fluid (e.g., a charged semi-dilute polymer solution or a charged polymer melt), a suspended conical droplet is formed, whereby the surface tension of the droplet is in equilibrium with the electric field. Electrostatic atomization occurs when the electrostatic field is strong enough to overcome the surface tension of the liquid. The liquid droplet then becomes unstable and a tiny jet is ejected from the surface of the droplet. As it reaches a grounded target, the jet stream can be collected as an interconnected web of fine sub-micron size fibers. The resulting films from these nanoscale fibers (nanofibers) have very large surface area to volume ratios and very small pore sizes.

The electrospinning technique was first developed by Zeleny [1] and patented by Formhals [2]. Up to now, there are about 50 patents on electrospinning technology. Much research has been done on how the jet is formed as a function of electrostatic field strength, fluid viscosity, and molecular weight of polymers in solution. In particular, the work of Taylor and others on electrically driven jets has laid the groundwork for electrospinning [3]. Although potential applications of this technology have been widely mentioned, which include biological membranes (substrates for immobilized enzymes and catalysts systems), wound dressing materials, artificial blood vessels, aerosol filters, clothing membranes for protection against environmental elements and battlefield threats [4-26], *no practical industrial process for electrospinning of polymer systems for fabric applications has ever been implemented*. The existing commercial electrospinning process by Donaldson, Inc. is limited for the manufacture of filter membranes, not of clothing. The major technical barrier for manufacturing electrospun fabrics for clothing is the speed of fabrication. In other words, as the fiber size becomes very small, the yield of the electrospinning process becomes very low. For example, if we consider a polymer melt being spun from the spinneret with a diameter of 700  $\mu\text{m}$  and the final filament is formed with a diameter of 250 nm, the draw ratio will then be about  $3 \times 10^6$ . As the typical throughput of the extrudate from a single spinneret is about 16 mg/min (or 1 g/hr), the final filament speed will be about 136 m/s, which is comparable to the highest speed (10,000 m/min or 167 m/s) attainable by the high-speed melt-spinning process. Thus, the throughput of the spinneret in electrospinning is about 1000 times lower than that in the commercial high-speed melt-spinning process.

### 1.2 Unique *e-Jets*<sup>TM</sup> Technology by STAR, Inc.

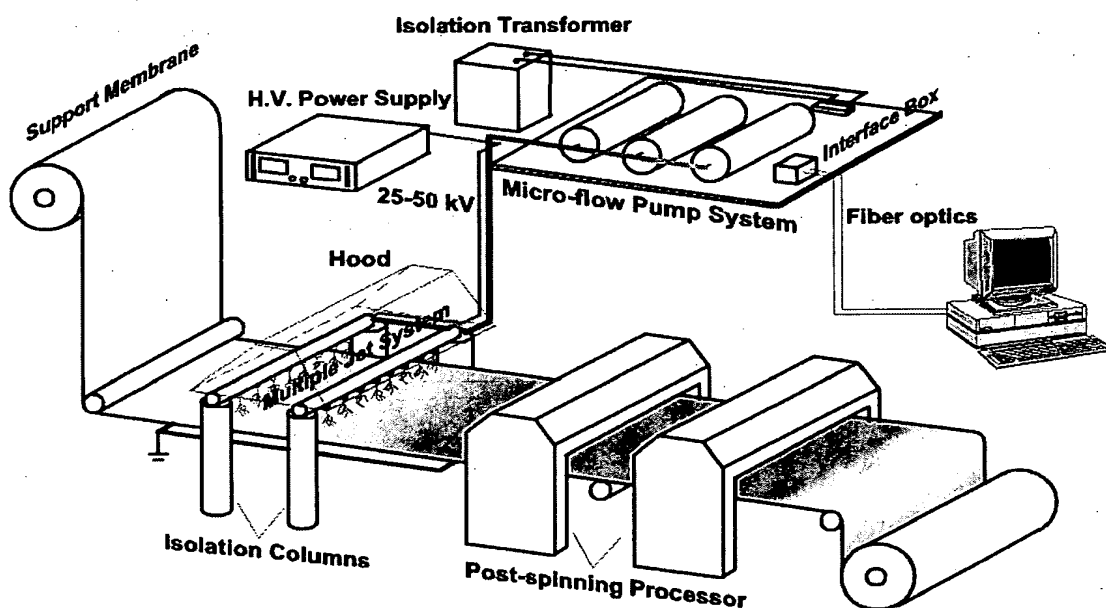
Another major technical problem for mass production of electrospun fabrics is the assembly of spinnerets during electrospinning. A straightforward multi-jet arrangement as in high-speed melt-spinning cannot be used because adjacent electrical fields often interfere with one another, making the mass production scheme by this approach very impractical.

A unique *e-Jets*<sup>TM</sup> technology for multiple-jet electrospinning process has recently been developed for manufacturing of non-woven membranes having fibers with diameters in the tens

## Unique Hybrid Multi-Jet Electrospinning and Melt-Blown Technology for Mass Production of Nanostructured Membranes

of nanometer size range by the PI from Stonybrook Technology and Applied Research (STAR), Inc. and scientists from the Chemistry Department in the State University of New York at Stony Brook (SUNYSB). Three patent applications based upon this technology have been filed in 2001 by the PI and Co-PIs through SUNYSB (1. "Control and Manipulation of Electrospinning Process"; 2. "Bioabsorbable Membrane for Prevention of Post-Operative Adhesions", 3. "Novel Bioabsorbable Scaffolds for Cell Delivery Applications"). The Technology Transfer Center at SUNYSB has agreed to grant exclusive licensing of these patents to STAR, Inc., when approved. A schematic diagram of the prototype multi-jet electrospinning production unit (with one-dimensional array electrodes), based on the *e-Jets™* technology and tailored for making nanostructured bioabsorbable membranes for biomedical applications, is shown in Figure 1. The unique features of this apparatus can be summarized as follows.

Fig.1 Bioabsorbable Nanostructured Membrane Processing Unit



1. Innovation in the *e-Jets™* technology:
  - Finite element analysis on electric field distribution of multiple spinnerets.
  - New spinneret/electrode designs for multiple-jet operation.
  - Electric field control on jet streams (formation and acceleration)
  - 3D pattern design.
2. Combination of materials and processing development permits us to develop new membrane materials including:
  - Variations in porosity, pore size, and pore size distribution
  - Variation in nanofiber/nanoparticle morphology
  - Variation in degree of hydrophobicity
  - Construction of unique hierarchical structures (nano-, micro-, meso-).

## Unique Hybrid Multi-Jet Electrospinning and Melt-Blown Technology for Mass Production of Nanostructured Membranes

We feel that, with some suitable modifications, the *e-Jets™* technology can be readily incorporated into the more conventional melt-blown process for manufacturing of new non-woven textile structures for fabrics, which forms the basis of this proposal.

### 1.3 Conventional Melt-Blown Process

The melt-blown process is a unique non-woven technology using high-velocity air to produce fibrous non-woven articles directly from polymer melts. This process is unique because it can be used to produce fibers with a large range of diameter (from 0.1  $\mu\text{m}$  to 10  $\mu\text{m}$ ). The basic technology was first demonstrated by the Naval Research Laboratory in 1950s and was incorporated into the commercial process by Exxon Chemicals (now ExxonMobil) in 1970s. Recently, there have been some renewal interests to develop new microfiber materials by 3M, Eastman Kodak, Kimberly-Clark, and Fleetguard Filter.

The typical melt-blown process consists of the several elements: extruder, metering pumps, die assembly, web formation, and winding. The die assembly is the most critical element, which has three distinct parts: polymer-feed distribution, die nosepiece, and air manifolds. The feed distribution is usually designed in such a way that the polymer distribution is less dependent on the shear properties of the polymer, allowing for the process of widely different polymeric materials. The die nosepiece is a tapered and hollow piece of metal having several hundred spinnerets across the width. The extruded polymer melt filaments are subsequently attenuated by hot air to form fine fibers. (The web uniformity depends largely on the design of the nosepiece.) The high velocity hot air (primary air) is supplied by the air manifolds through the slots on the top and bottom sides of the die nosepiece. Typical air temperature ranges from 200°C to 350°C at velocity ranges from 0.5 to 0.7 times of the speed of sound. As the primary air stream containing the microfibers progresses toward the collector screen, it draws in a large amount of surrounding air (secondary air) that cools and solidifies the fibers. The fibers are generally laid randomly on the moving collector (usually a vacuum is applied to the inside). The collector speed and the collector distance from the die nosepiece can be varied to produce a variety of non-woven morphology.

### 2. Phase I Technical Objectives

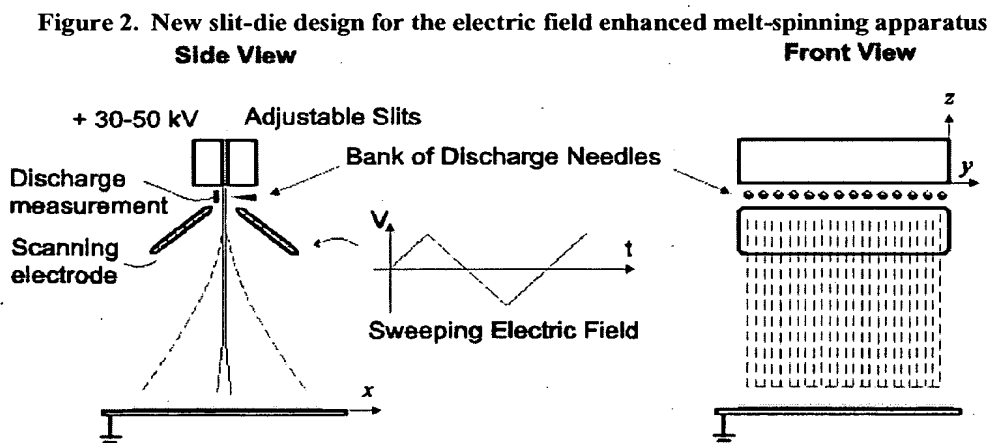
This SBIR Phase I Project aims to develop a new form of technology that will combine the melt-blown process with the *e-Jets™* (multiple-jet electrospinning) technology. The key technology should permit fabrication of membranes with composite microfiber/nanofiber hybrid morphology and can lead to the production of nanostructured composite materials in new formats.

The specific aims of this Phase I proposal are to implement new designs in electrospinning/electrospraying technology in order to incorporate the *e-Jet™* technology (patent pending) with the more conventional melt-blown process. Complex and coupled processing parameters including novel spinneret assemblies, new electrode designs, and control of jet acceleration, transportation and manipulation will be considered.

### 3. Phase I Work Plan

### 3.1 Design 1 – Linear Electrical Field Enhanced Melt Spinning Assembly

Based on our unique *e-Jets™* technology for electrospinning of polymer solutions, we propose to develop a prototype multi-jet apparatus for electrospinning of polymer melts. The conceptual design of a linear electrical field enhanced melt spinning assembly is shown in Figure 2, where a relatively high throughput of polymer melt (about 1 gm/min/spinneret) can be extruded from a pair of adjustable slits. The slit exit surfaces will contain microchannels with defined dimensions and separation distances. The presence of microchannels will provide the exit for fiber spinning. The slits are connected to the high voltage. The shape and separation distance of the spinning “holes” will be designed using a finite element analysis to simulate multi-jet electrospinning process, which will be described later. Under the electric field, the charged molecules in the melt will be stretched out and form bundles of repelling fibers in the flight path to the target plate. Different morphology in the membrane may be formed by tuning the spinning speed from the die. The amount of the electric charge in the polymer solution (excess charge) can be controlled by electric charge “spray”. In this case, a bank of discharge needles will be connected to another high voltage power supply and the charges in the corona zone of the needle tips will be transferred to the polymer melt. By utilizing this technique, 75% - 100% of excess charge may be obtained. A pair of steering (scanning) electrodes will be placed at the down stream but located very close to the bank of the discharge needles. By applying a sweeping voltage on to the scanning electrodes, a membrane with a controlled pattern formation can be obtained. The advantages of this proposed technique are: (1) relatively high throughput (high yield) – 1 to 2 orders of magnitude higher than the electrospinning of polymer solutions (2) no moving parts.



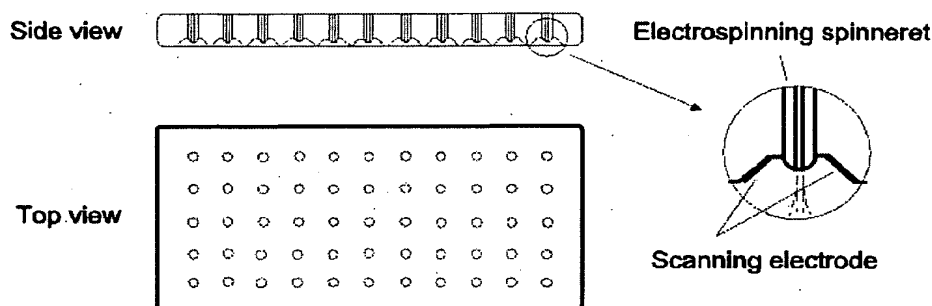
### 3.2 Design 2 – Two-Dimensional Multi-Jet Assembly

To explore the possibility to further increase the total electrospinning throughput, without the concerns of electrical field interference between the spinnerets, we propose to develop a prototype two-dimensional array of electrospinning spinnerets in an isolation matrix. This design is quite different from Design 1 where secondary electrodes were used to insulate the electrical field of each spinneret. The conceptual design of this apparatus is shown in Figure 3, where each spinneret has two pairs (X and Y direction) of miniature scanning electrodes. The

### Unique Hybrid Multi-Jet Electrospinning and Melt-Blown Technology for Mass Production of Nanostructured Membranes

spinneret and the scanning electrodes are constructed in a way, based on the electrical field calculation, to minimize the interference between the adjacent electrodes. They are also electrically wired such that each individual polymer solution jet can be turned on and off and be steered to a finite size target area. As each spinneret can be turned on/off independently by electricity (the response time thus should be relatively fast) a designed pattern can be obtained in the resultant membrane.

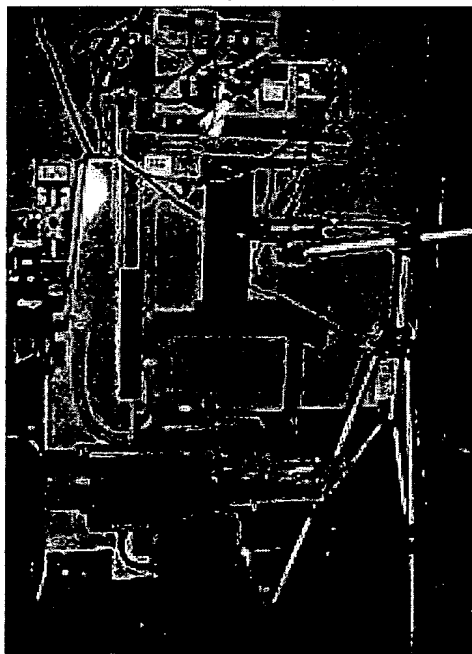
Figure 3 New designed array-spinneret pattern for electrospinning



Both Design 1 and Design 2 prototype multiple-jet electrospinning assemblies will be tested in a laboratory size portable melt spinning apparatus (photograph is shown in Figure 4). (This equipment was originally designed for on-line X-ray and Raman studies of melt-spinning process.) This apparatus consists of a 3/4" Independent Laboratory single screw extruder (C. W. Brabender Instr. Inc., NJ) and a custom-built vertical lifter with about 1.2 m of displacement (Applied Automation Research Corp., FL). The maximum extrusion temperature is about 325 °C. The extruder is mounted on a horizontal platform that can be translated in the vertical direction by computer control. The range of the typically used mass output rate of this extruder is 1-7 g/min.

The advantages of Design 1 (Linear Electrical Field Enhanced Melt Spinning Assembly) include the easy fabrication of the spinneret assembly, easy operation and maintenance, higher throughput rates. The disadvantages of this design include the difficulty in isolating the electric field distribution for each spinneret. In contrast, the advantages of Design 2 (Two-Dimensional Multi-Jet Assembly) include that the electric field of each spinneret will be isolated and that a more uniform fiber diameter and membrane morphology should be feasible. However, the disadvantages of this design are that the production and the operation of the spinning assembly may be more difficult. As both designs of the multi-jet assemblies will be tested, the one that provides the best

Figure 4. Portable extrusion apparatus at Stony Brook



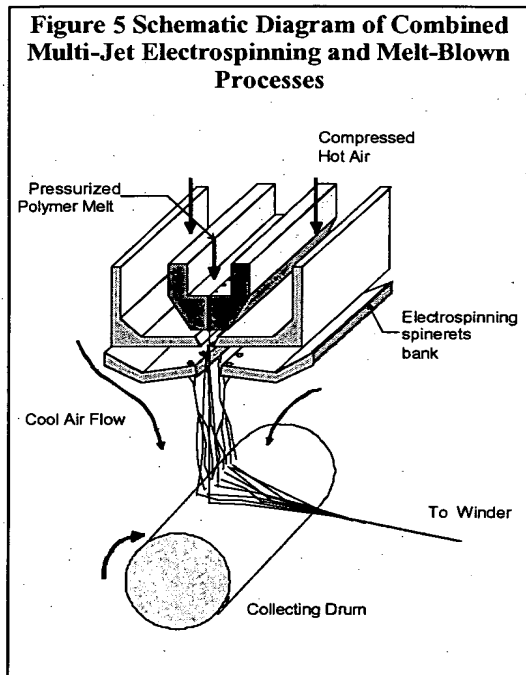


## Unique Hybrid Multi-Jet Electrospinning and Melt-Blown Technology for Mass Production of Nanostructured Membranes

performance will be incorporated with a laboratory scale melt-blown apparatus, whose scheme will be outlined next. Depending on the results from the experimental testing of both designs, we will also consider the possibility of a hybrid design by combining some unique features from each design.

### 3.3 Combination of Multi-Jet Electrospinning Technology and Melt-Blown Process

We propose to incorporate the best performing multi-jet electrospinning assembly in a laboratory melt-blown spinning apparatus using a slit-die or a pin-hole geometry. We term this *simultaneous co-spinning* design. The pin-hole assembly can be manufactured at SUNY-Stony Brook. The construction of the slit die assembly (polymer-feed distribution, die nosepiece, and air manifolds) for the melt-blown process can be contracted to the Hills, Inc. (West Melbourne, Florida), which is one of the leaders in the production of melt-blown manufacturing equipment. The feed distribution will be designed to minimize the shear properties of the polymer, allowing the melt blowing of widely different polymeric materials. We will probably choose the coat-hanger type feed distribution system because it gives both even polymer flow and even residence time across the full width of the die. The membrane uniformity depends largely on the design and fabrication of the nosepiece. Therefore, we will require very tight tolerances for the die nosepiece. There are two types of die nosepiece: capillary type and drilled holes type. We will probably choose the capillary type because the problems associated with precise drilling of very small holes can be avoided. In addition, the capillary tubes can be precisely aligned so that the holes follow a straight line accurately. The testing materials for the proposed study will be metallocene based isotactic polypropylene (iPP) of different molecular weights. This material is commonly used in melt-blown fabrics.



A conceptual *simultaneous co-spinning* design to combine the multi-jet electrospinning technology and the melt-blown process is illustrated in Figure 5. In this design, the electrodes (electrospinning spinnerets) are aligned at a tilted angle with respect to the melt-spinning axis. The zone of instability of the jet will be immersed with the high velocity primary air, allowing the charged fibers to be extended and entangled with the melt-blown fibers. Additional air streams will also be applied in the down stream of the spin line to enhance the fiber mixing and to facilitate the fiber collection. The combined effects of electrostatic repulsions and the high velocity of the air stream will be utilized to create a new type of nanofiber morphology. In order to produce a 1-m width fabric, we plan to construct a large moving platform (ground electrode) for sample collection.

## Unique Hybrid Multi-Jet Electrospinning and Melt-Blown Technology for Mass Production of Nanostructured Membranes

In this study, we have set a target ratio of 10% nanofibers (from electrospinning) and 90% microfibers (from melt-blown process) in the initial production of the non-woven fabric. We know that the surface area of this 10% nanofibers is about 10 times more than that of the 90% microfibers produced by the melt-blown process. Since the typical throughput from a single spinneret in electrospinning is about 1000 times smaller than that in the melt-blown spinning process, we plan to take the following steps to reach this goal. (1) The diameter of the melt-blown spinneret will be reduced and the air velocity will be reduced to the lowest value (such as 0.2 times of the speed of sound). This should reduce the throughput of the melt-blown spinning by more than 50 times. (2) A ratio of 10 spinnerets in electrospinning to 1 spinneret in melt-blown spinning will be implemented. This will increase the throughput of electrospinning by 10 times. These two steps should allow us to produce the fabrics with a nanofiber/microfiber ratio close to 10%. If necessary, we can further increase the content of the nanofibers by coating the simultaneously co-spun product using a sequential electrospinning method, as illustrated in Figure 1. We note that, the sequential electrospinning process can produce an asymmetric layered product.

### 3.4 Performance Evaluations of Hybrid Microfiber/Nanofiber Membranes

As requested by the Natick Soldier Center of this SBIR grant, we plan to demonstrate the co-spinning technology (melt blown and multi-jet electrospinning) that can be scaled up to manufacture a 1-meter width fabrics. Scientists from the Natick Soldier Center will test the following properties of the melt-blown/electrospun fabrics, under the guidance of or with the assistance. These properties will include (1) water vapor transport through the fabric, (2) air penetration through the fabric, (3) liquid (oil and water) retention within the fabric, and (4) liquid contact angle. The properties (air resistance, breath ability, water retention and liquid contact angle) will be optimized by fine-tuning the multiple operating parameters in the multi-jet electrospinning process. The routine characterizations of these membranes such as scanning electron microscopy (to study the membrane morphology), thermal analysis (to study the thermal properties such as melting and glass transition temperatures), mechanical properties, and X-ray scattering/diffraction (to study crystal structure and lamellar morphology) will also be carried out on an as-needed basis.

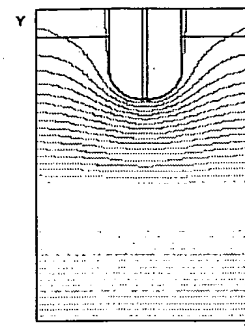
## 4. Related Work

In the past three years, we have successfully developed a unique *e-Jets*<sup>TM</sup> technology (patent pending and *TM* being processed) at *STAR*, Inc. and at SUNY-Stony Brook. The key technology permits fabrication of membranes with composite nanofiber/nanoparticle hybrid morphology, designed composition variations, and 3D pattern formation from polymer solutions. This technology shall enable us to design a new multi-jet apparatus for melt-spinning that can also be incorporated in the melt-blown process to produce new microfiber/nanofiber composite non-woven fabrics. We will briefly outline the principle of the *e-Jets*<sup>TM</sup> technology and the innovation as follows.

### 4.1 Finite Element Analysis to Simulate Multi-Jet Electrospinning Processes

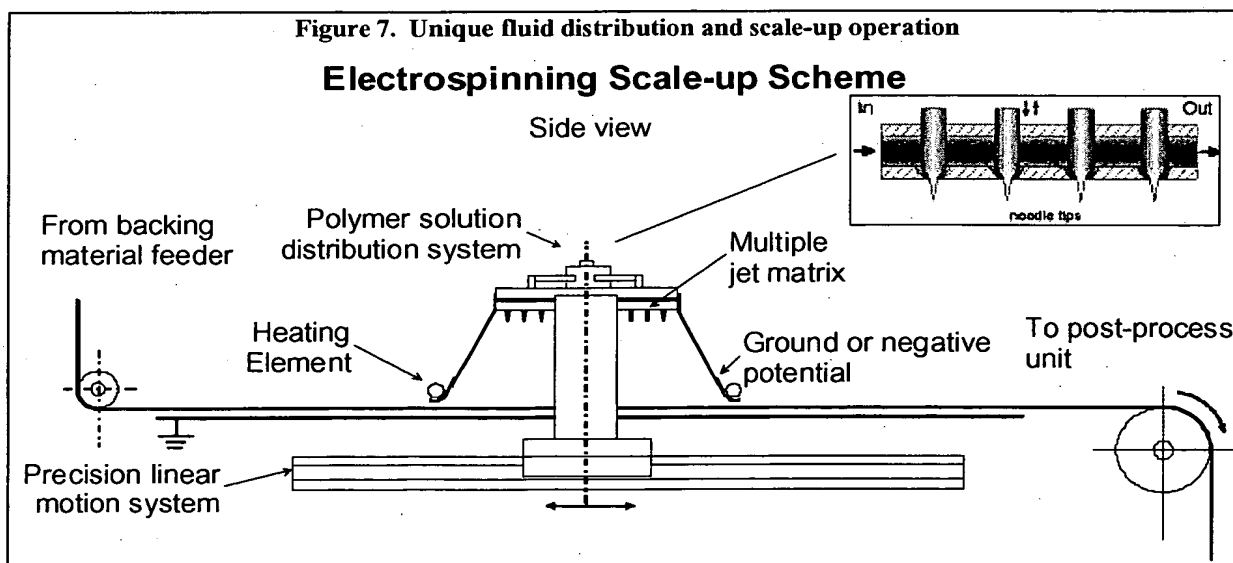
## Unique Hybrid Multi-Jet Electrospinning and Melt-Blown Technology for Mass Production of Nanostructured Membranes

In the multi-jet electrospinning system, the electric field distribution is very complicated. The design of the multi-jet electrode array in the current apparatus was guided by the 2D finite element analysis (FEA) for electromagnetics using the software by Field Precision (www.fieldp.com). The software package included XLATE, MESH, ESTAT and VESTAT modules, which were used to numerically analyze the electric field distributions in complex electrode configurations. Extensive simulations for a single jet system involving parameters such as size, shape and potential of the electrode, distance between the electrode tip and the ground, the shape and materials surrounding the electrode were carried out (Figure 6). We have constructed several electrodes based on different designs and experimentally verified the electrode designs with the FEA simulations. The parameters obtained from simulations and experiments for the single jet were also used as basis for the design of the multi-jet system.



### 4.2 Polymer Fluid Distribution System for Multi-jet Electrospinning

Figure 7 shows a schematic diagram of the fluid distribution and the linear array electrode assembly of a prototype scale-up apparatus. The backing material for the membrane is fed into the system by a “convoy belt” method. The polymer solution is distributed to the multiple spinneret (up to 200) linear array system with minimum pressure drop (inset diagram). The array system is mounted on two electrically isolated posts that are seated on a pair of precision rails. This allows the array system to move along the “belt” direction back and forth. The precision rails can also be mounted on a “rocking” system so that the array can move in the direction perpendicular to the “belt” direction. The heating elements are implemented to control the solvent evaporation rate. The “belt” can be sent to another unit or a post-processing unit for manufacturing composite membranes.



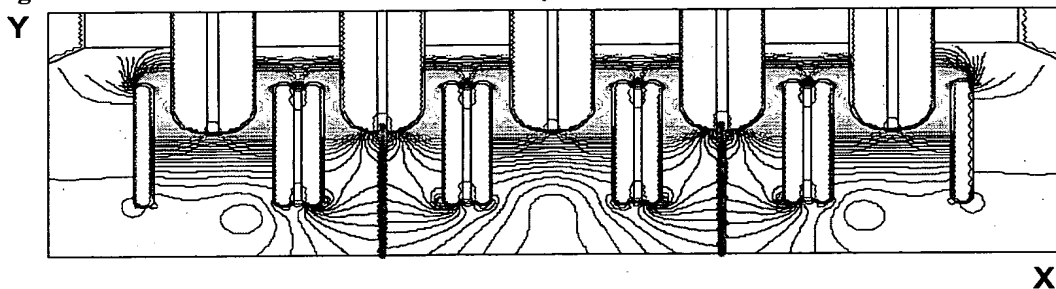
### 4.3 New Innovative Designs for The *e-Jets*<sup>™</sup> Technology

Several new and innovative schemes to improve the control and manipulation of the electrospinning process have been implemented in order to control more precisely the fiber diameter and the membrane morphology. These methods are based on a combination of the principles of plasma physics and accelerator physics. They address two distinct processing issues in electrospinning: (1) the process for the jet formation and (2) the process for the jet acceleration. The principles of jet formation in an electrospinning process are similar to those of plasma formation in an “ion source”.

#### 4.3.1 Use of Secondary and Tertiary Electrodes to Optimize Multi-jet Spinning

The use of secondary electrodes to shield each primary electrode (spinneret) has been implemented in our prototype apparatus. We note that the presence of secondary electrodes can weaken the field strength at the electrode tip. To overcome this problem, the geometrical shape, location and electric potential of the secondary electrodes has been optimized by the FEA simulations. We have optimized the designs with the parameters for single jet operation as a reference for multi-jet designs. The following two criteria have been met simultaneously in the design: (1) each electrode in the multi-jet system has the same electric field distributions, (2) the electric field strength on the electrode tip in the multi-jet system is the same as that in the single jet system. We have paid special attention to the field strength change before and after the jet formation. Figure 8 illustrates the simulation of electric field distribution for a 5-electrode system with two jets being formed. Results suggest that the interference of the neighboring electrodes (with and without the jet formation) of the optimized system is about 1% using the secondary electrodes. A real multi-jet system has been constructed based on the optimal parameters obtained from detailed FEA simulations. This analysis will also be carried out to optimize Design 1 and Design 2 of the multiple-jet electrodes assembly for melt spinning.

Figure 8. The FEA calculation of a 5-electrode system with formation of only two polymer jets.



We have also used a tertiary electrode to facilitate the membrane collection process during electrospinning. The tertiary electrode can be placed very close to the surface of the membrane. The electrode is connected to the ground with a negative potential in order to remove the surface charge accumulated by the electrospinning process efficiently.

#### 4.3.2 Variation of Electrical/Mechanical Properties of Conducting Fluid

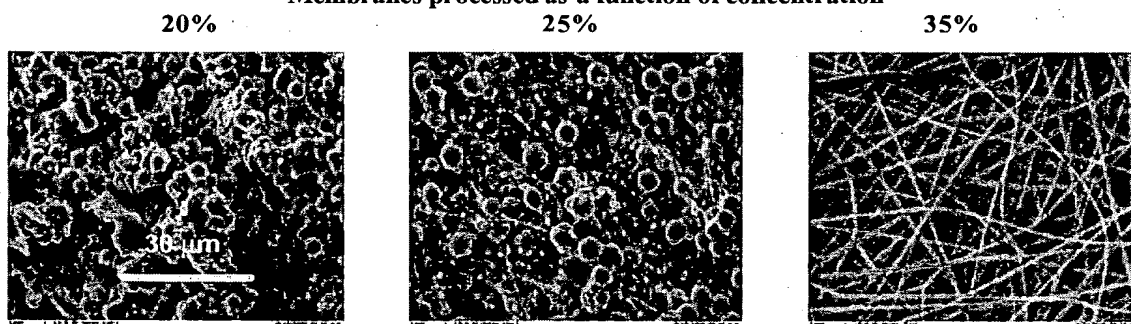
## Unique Hybrid Multi-Jet Electrospinning and Melt-Blown Technology for Mass Production of Nanostructured Membranes

The conductivity of the macromolecular solution can be drastically changed by adding ionic inorganic/organic compounds. The magneto-hydrodynamic properties of the fluid depend on a combination of physical and mechanical properties, (e.g., surface tension, viscosity and viscoelastic behavior of the fluid) and electrical properties (e.g., charge density and polarizability of the fluid). For example, by adding a surfactant to the polymer solution, the fluid surface tension can be reduced, so that the electrostatic field can influence the jet shape and the jet flow over a wider range of conditions. By coupling a pump system that can control the flow rate either at constant pressure or at constant flow rate, the effect of viscosity of the conducting fluid can be alleviated.

The manipulation capability through processing parameters provides not only the means to control the fiber diameter, the membrane porosity but also the fiber/particle morphology of the membrane. The following process parameters have been explored for this purpose.

	Parameter	Methods
1. Fluid	Viscosity	Use of miscible solvents; polymer concentration
	Dielectric constant	Use of miscible solvents
	Surface tension	Addition of surfactants
	Viscoelastic property	Molecular weight/polydispersity and concentration
	Charge density	Change in ionic strength and pH; use of mixtures
	Polarizability	Use of miscible solvents
2. Fluid flow	Flow rate	Constant pressure or constant flow rate
3. Jet formation	Nozzle effect	Nozzle geometry
	Electrostatic potential	Control of field strength and field geometry
4. Jet acceleration	Electrostatic potential	Control of field strength and field geometry along the flight path, use of alternating gradients.

**Figure 9. SEM image of electrospun PLGA membranes with different morphology**  
Membranes processed as a function of concentration



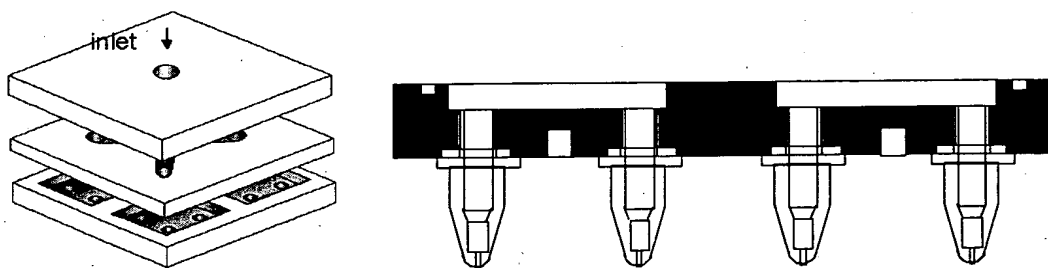
For example, we have investigated the effect of charge density (through the addition of salts) on the fiber diameter [27]. We found that the fiber diameter could be significantly changed by adding a small amount of salt in the solution. When 1 wt% potassium phosphate ( $\text{KH}_2\text{PO}_4$ ) was added to the biodegradable poly(lactide-co-glycolide) PLA-co-PGA solution, the fiber diameter became much thinner than the one with no salt added. Thus, higher excess charge density generally favors the production of thinner fibers and lower excess charge density favors

the production of thicker fibers. Several other kinds of salts (e.g. NaCl,  $\text{KH}_2\text{PO}_4$ ,  $\text{KIO}_3$ , and  $\text{K}_3\text{PO}_4$ ), which are all biologically compatible to the body, will also be considered. We have demonstrated that the membrane morphology could be controlled by different processing parameters. By tuning the concentration, flow rate and/or solution viscosity, a membrane with different morphology (nanofiber to nanoparticle) could be obtained (Figure 9).

#### **4.3.3 New Electrode Design and 2D Multi-jet Array Assembly for Solution Spinning**

The designs of the electrospinning electrode have been intended to separate the jet formation process and the jet acceleration process using the following principle. The jet formation was treated as an “ion source”; the positively charged electrode was treated as the ‘anode’. The anode was responsible for the formation of the polymer solution droplet while the ‘cathode’ was a plate electrode with a small exit hole in the center. This exit hole provided the means to let the jet stream to pass through the cathode. If we take the polymer droplet on the anode to have a typical dimension of 2~3 mm and place the cathode at a distance of about 10 mm from the anode, a reasonable electrostatic potential can be developed. The short distance between the two electrodes implies that the electrostatic potential could be fairly low. However, the resultant electric field strength could be sufficiently strong for the electrospinning process. By varying the electric potential of the anode, the jet formation could be controlled and adjusted. Such an electrode configuration has greatly reduced the required applied potential on the anode from about 15 kilovolts (kV) down to typically 1.5 to 2 kV (relative to the cathode base potential). The anode potential required for a stable jet formation depends on the electric/mechanical properties of the conducting fluid.

Different from the linear array electrode assembly, a prototype two dimensional multi-jet system with 4x4 electrode/spinneret array assembly has been designed and constructed as shown in Figure 10. The polymer solution was introduced by a central inlet and then distributed to four quadrant sub-compartments. The electric potential  $V_0$  was applied to the electrodes and another potential  $V_1$  was applied to the secondary electrodes made by a metal plate. Experimental results showed that interference effects were low as expected. We are in the process of optimizing the parameters controlling the fluid flow and the electric field distributions for this system. Our target system in this proposal will be a 10x10 multi-jet system using the designs of primary, secondary and tertiary electrodes.



**Figure 10. 3D view of the solution distribution system and the spinnerets mounting diagram.**

#### **4.3.4 Control of Jet Acceleration, Transportation and Manipulation**

## Unique Hybrid Multi-Jet Electrospinning and Melt-Blown Technology for Mass Production of Nanostructured Membranes

The jet stream from the cathode exit hole is a stream that is positively charged. This stream has a tendency to straighten itself during flight. However, without the external electric field confinement, the jet will soon become unstable in its trajectory. In other words, the charged beam becomes defocused and thus the jet stream, with the intrinsic bending instability due to electrostatic repulsion of the charges within the jet stream, could not provide the desired function in controlling the microscopic and macroscopic properties of the fluid. This instability can be partially removed by using carefully designed probe electrodes immediately after the cathode plate and a series of (equally) spaced plate electrodes. The electrode assembly (i.e., the probe electrode and the plate electrodes) can create a uniform distribution of electrostatic potential along the (straight) flight path. The acceleration potential is formed by placing the base potential of the ion source at about +20 to +30 kV above the target (at ground potential) while the electrostatic potential of the probe electrode can be adjusted to slightly below the cathode base potential. The composite electrodes are capable to partially stabilize the jet stream.

The jet stream can also be focused by using an “Alternating Gradient” (AG) technique, widely used in the accelerator technology of high-energy physics. The basic idea is to use two pairs of electrostatic quadrupole lenses. The second lens has the same geometric arrangement as the first lens with a reversed (alternate) electric gradient. The beam will be focused, for example, in the  $xz$  plane after the first lens and then be refocused in the  $yz$  plane after the second lens. By applying an additional triangle-shaped waveform to the potential on one of the pairs of the quadrupole, the jet can be swept across the target area, allowing the control of the direction of the jet stream. Furthermore, with varying waveform of the ‘sweep’ potential, a desired pattern on the target can be formed by using this technology.

The combination of above four schemes (4.3.1-4.3.4) form the basis of the unique *e-Jets™* technology.

### 5. Research Milestones (6 months Phase I + 4 months Option Period) and Future Research and Development

The research milestones for the first 6 months of this Phase 1 project and the second 4 months of the Option Period are summarized below.

	<i>Month1-3</i>	<i>Month4-6</i>	<i>Month 7-8</i>	<i>Month 9-10</i>
<i>Design 1 Construction and Testing</i>	<b>X</b>			
<i>Design 2 Construction and Testing</i>	<b>X</b>			
<i>Combination of Melt-Blown Process with Multiple-Jet Electrospinning</i>		<b>X</b>		
<i>Membrane Property Evaluations (air resistance, breathability, water retention and liquid contact angle)</i>			<b>X</b>	<b>X</b>
<i>Optimization of Designs and Processing Variables</i>			<b>X</b>	<b>X</b>

The successful operation of this Phase I project will include (1) the demonstration of a multiple-jet electrospinning unit that can be scaled-up and incorporated in the conventional melt-

## Unique Hybrid Multi-Jet Electrospinning and Melt-Blown Technology for Mass Production of Nanostructured Membranes

blown process, (2) the fabrication of a new class of non-woven materials containing microfiber/nanofiber hybrid morphology, which possesses superior liquid and vapor transport properties. If successful, the technology base developed in Phase I will be fully extended to develop a commercially viable system with scale-up capability. At that point, a potential joint venture with ExxonMobil or other willing partner will be explored.

### 4. Commercialization Strategy

If successful, we plan to incorporate the best performing multi-jet electrospinning assembly in a commercial melt-blown spinning facility to evaluate the scale-up capability in the Phase II study. We will try to first secure the intellectual right at STAR, Inc. and then to explore a partnership relationship with a commercial outfit that is specialized in the melt-blown technology. One such company may be the Baytown Polymer Center, ExxonMobil Chemical Company. One of the Co-PIs (Ben Hsiao) is currently a consultant with ExxonMobil and has been conducting the research project on "Flow-Induced Crystallization in Polyolefins" with them since 1998. Currently, ExxonMobil is the leader in the melt-blown technology development, holding most of the licenses and/or options to produce microfiber non-woven and melt-blown equipment. We will also consider other companies such as 3M, Eastman Kodak, Kimberly-Clark, and Fleetguard Filter for potential partners.

### 5. Key Personnel (PI and Co-PIs)

*Stonybrook Technology & Applied Research, Inc., (STAR)* was established by three scientists (Benjamin Chu, Benjamin S. Hsiao and DuFei Fang) in 1999. They aim to take advantage of their complimentary knowledge in chemistry, materials science and engineering, and physics to (1) develop unique technologies and (2) produce specific cost-effective products, made of polymers and nano-composites. Their combined expertise includes chemical knowledge on polymer synthesis, physical processing methods, as well as advanced technology on molecular structure and macroscopic property relationships that permit predictions on a range of specific functional properties of materials. The initial emphasis of *STAR* will be on the development of electrospinning technology for applications to biomedical products dealing with anti-adhesion membranes, cell delivery and pain management. The qualifications and the responsibility of PI/Co-PIs for this proposal are summarized below.

Dufei Fang, Ph.D. (Principal Investigator) is the Technology Director of Stonybrook Technology and Applied Research, Inc. (STAR). He is an experimental physicist with background in accelerator physics and plasma physics. He has extensive experience in the instrumentation design and construction for free electron laser and synchrotron X-ray facilities. He has designed a prototype electrospinning apparatus with commercial scale-up possibility. DF was a professor and group leader at the Institute of Modern Physics, Fudan University (China) and also a visiting Fellow at Fitzwilliam College, Cambridge University (U.K.). He will be responsible for instrumentation implementation and optimization of the electrospinning process.

Ben Chu, Ph.D. (Co-PI) is a Distinguished Professor with joint appointment in both the Chemistry Department and the Department of Materials and Engineering at Stony Brook with specialization in hydrogels, polyelectrolyte, surfactant complexes, nanostructure modifications,



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ion-containing polymers, colloid science and DNA capillary electrophoresis. A High Polymer Physics Prize winner, BC is an executive member of the State University of New York (SUNY) Beam line at X3, a Co-Spokesperson for the Advanced Polymers Beam line (APB) at X27C of National Synchrotron Light Source (NSLS), and is serving as one of the Co-PIs in the ChemMat CARS at the Advanced Photon Source. He will be responsible for the studies of structure/property/process relationships of the electrospun membranes from polymer melts.

Ben Hsiao, Ph.D. (Co-PI), a Professor in the Chemistry Department at Stony Brook, came from the Fibers and CR&D Departments at DuPont and has extensive synchrotron experience and research expertise in the area of structure, morphology, property, functionality and processing relationships in biodegradable polymers, suture fibers and nanocomposites. BH is the Spokesperson for the APB at X27C of NSLS. He will be responsible for incorporation of the multiple-jet electrospinning prototype apparatus with the melt-blown process. BH is a long-time consultant with ExxonMobil and has several on-going research projects on fiber spinning with them.

### **6. Facility and Equipment**

The STAR, Inc. occupies 600 square ft of space in the Chemistry Building at SUNYSB. Through the Chemistry Department, researchers can access scanning electron microscope (SEM), transmission electron microscope (TEM), scanning transmission X-ray microscope (STXM), Gamma-irradiation facilities, HRTEM and the synchrotron X-ray scattering beamlines (X3A2, X27C) in the National Synchrotron Light Sources (NSLS), Brookhaven National Laboratory. At Stony Brook, melt-spinning and solution spinning devices, atomic force microscope, (AFM) several high-resolution nucleus magnetic resonance (NMR) instruments (13C-, 1H-), differential scanning calorimetry (DSC), Fourier Transform Infrared (FTIR), Raman Spectroscopy, tensile testing machines and rheometers are also available. Other capabilities include a rotating anode X-ray generator, forced Rayleigh scattering, centrifuge ball viscometry, magnetic needle rheometry, and DNA capillary electrophoresis. Machine shop is also available..

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1994 - 1997 Professor of Physics, Associate Director of Accelerator Laboratory, Fudan University, China

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1991 summer, Visiting Scientist, Dept. of Nuclear Engineering, University of Illinois at Urbana/Champaign; 1993

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**Publications (1997 - 2001):**

HB Gao, DF Fang, FQ Lu et al, "Electron impact ionization cross section of Ar<sup>+</sup>, Kr<sup>+</sup>, In<sup>+</sup> and Ge<sup>+</sup>", Nucl. Instr. & Methods B132, 364 (1997)

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### Employment History:

Assistant Professor of Chemistry, University of Kansas 1962-1965; Associate Professor of Chemistry, University of Kansas 1965-1968; Professor of Chemistry, SUNY at Stony Brook 1968-1988; Chairman, 1978-1985; Professor of Materials Science and Engineering, 1982- ; Leading Professor of Chemistry, SUNY/Stony Brook 1988-1992; Distinguished Professor, SUNY/Stony Brook 1992-

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### Editorial Boards:

Associate Editor, Materials Letters, 1986-1989; Editorial Board, Journal of Colloid and Interface Science, 1986-1989; Editorial Advisory Board, Macromolecules, 1990-1992; Editorial Board, Review of Scientific Instruments, 1993-1995; Editorial Advisory Board, Journal of Polymer Science, Part B (Polymer Physics), 1990-

### Fellowships, Special Invitations and Honors:

Participant of the 1966 Study Week on Molecular Forces, Pontifical Academy of Science, Vatican City, Rome, Italy; Alfred P. Sloan Research Fellow, 1966-1968; John Simon Guggenheim Fellow, 1968-1969; Visiting Professor, Japan Society for the Promotion of Science (JSPS), 1975-1976, 1992-1993; Humboldt Award for Senior U.S. Scientists, 1976-1977, 1992-1993; Distinguished Achievement Award in Natural Science, St. Norbert College, 1981; Fellow, American Institute of Chemists; Fellow, American Physical Society; Honorary Professor of the Chinese Academy of Sciences, 1992- ; High Polymer Physics Prize, American Physical Society, 1993; Langmuir Distinguished Lecturer Award, Division of Colloid & Surface Chemistry, American Chemical Society, 1994.; Honorary Professor, Nankai University, 1996- ; Award for Distinguished Service in Advancement of Polymer Science, Society of Polymer Science, Japan, 1997; Honorary Professor, Xiamen University, 1998- ; Outstanding Achievement Award, Chinese Institute of Engineers/USA, 1998.

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### Five Relevant Publications:

Shaofeng Ran, Christian Burger, Dufei Fang, Xinhua Zong, Sharon Cruz, Benjamin Chu, Benjamin S. Hsiao, Robert A. Bubeck, Kazuyuki Yabuki, Yoshihiko Teramoto, David C. Martin, Michael A. Johnson and Philip M. Cuniff, "In-situ Synchrotron WAXD/SAXS Studies of Structural Development during PBO/PPA Solution Spinning," *Macromolecules*, 35, 433-439 (2002).  
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Phillips, "Structural and Morphological Studies of Isotactic Polypropylene Fibers during Heat/Draw Deformation by in-Situ Synchrotron SAXS/WAXD," *Macromolecules*, **34**, 2569-2578 (2001).

### Five Additional Related Publications:

Benjamin S. Hsiao and Benjamin Chu, "Scattering: Light, Neutrons and X-Rays," in *Encyclopedia of Chemical Physics and Physical Chemistry, Vol. II: Methods*, eds. J. H. Moore and N. D. Spencer, IoP Publishing Ltd., Philadelphia, PA, Section B1.9, pp.1197-1225 (2001).

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Consultant: DuPont, Dow Chemical, Ethicon Inc., ExxonMobil, Fellowes

Program Committee for APS Workshop on Polymer Scattering (1994), NSLS User Workshop (1996), Denver X-ray Conference (1997), ACS-PMSE Symposium "Scattering from Polymers" (1998, 2001), Chair, SAS-SIG, ACA (1999), XI International SAS Conference at Brookhaven National Lab (1999)

Professional Societies: American Chemical Society; American Crystallographic Association; American Physical Society; Materials Research Society; American Association for the Advancement of Science

**Honors and Awards:**

Sigma Xi; Phi Kappa Phi; University of Connecticut Doctoral Fellowship (1985); Two SPE-ANTEC Best Technical Papers (1992); DuPont Young Faculty Award (1998-2000); Guest Professor in Changchun Institute of Chemistry, Chinese Academic of Sciences

**B. Publications.**

(134 referred publications, 20 pending publications, 85 conference proceedings, 6 patent applications and 1 book)

**Five Publications Most Related to the Project:**

- b. J. M. Samon, J. M. Schultz, B. S. Hsiao, S. Seifert, N. Stribeck, I. Gurke, C. Saw and G. Collins, "Structure Development during the Melt Spinning of Polyethylene and Poly(vinylidene) Fibers by in-situ Synchrotron Small- and Wide-Angle X-ray Scattering Techniques", *Macromolecules*, 32(24), 8121-8132 (1999).
- c. S. Ran, D. Fang, S. Zong, B. S. Hsiao, B. Chu, and P. M. Cunniff, "Structural Changes during Deformation of Kevlar Fibers via On-Line Synchrotron SAXS/WAXD Techniques", *Polymer*, 42(4), 1601-1612 (2000).
- d. R. H. Somani, B. S. Hsiao, A. Nogales, S. Srinivas, A. H. Tsou, I. Sics, F. J. Balta-Calleja, and T. A. Ezquerro, "Structure Development during Shear Flow Induced Crystallization of i-PP: In-situ Small Angle X-ray Scattering Study", *Macromolecules*, 33(25), 9385-9394 (2000).
- e. Bruce X. Fu, Ling Yang, Rajesh H. Somani, Steven X. Zong, Benjamin S. Hsiao, Shawn Phillips, Rusty Blanski and Patrick Ruth "Crystallization Studies of Isotactic Polypropylene Containing Nanostructured Polyhedral Oligomeric Silsesquioxanes (POSS) Molecules under Quiescent and Shear Conditions", *J. Polym. Sci. Polym. Phys.*, 39(22), 2727-2739 (2001).
- f. S. Ran, X. Zong, D. Fang, B. S. Hsiao, B. Chu and R. A. Phillips, "Structural and Morphological Studies of Isotactic Polypropylene Fibers during Heat/Draw Deformation by in-situ Synchrotron SAXS/WAXD", *Macromolecules*, 34(8) 2569-2578 (2001).

**Five Additional Significant Publications:**

- B. S. Hsiao, B. B. Sauer, R. Verma, B. Chu, P. Harney, H. G. Zachmann and S. Seifert, "New Insight of Isothermal Melt Crystallization Via Time-Resolved Simultaneous SAXS/WAXD Measurements", *Macromolecules*, 28, 6931-6936 (1995).

# **Unique Hybrid Multi-Jet Electrospinning and Melt-Blown Technology for Mass Production of Nanostructured Membranes**

- L. Zhu, S. Z. D. Cheng, B. H. Calhoun, Q. Ge, R. P. Quirk, E. T. Thomas, B. S. Hsiao, F. J. Yeh and B. Lotz, "Crystallization Temperature-Dependent Crystal Orientations within Nanoscale Confined Lamellae of a Self-Assembled Crystalline-Amorphous Diblock Copolymer", J. Am. Chem. Soc., 122(25), 5957-5967 (2000).
- Z. G. Wang, B. S. Hsiao, E. B. Sirota, P. Agarwal and S. Srinivas, "Probing the Early Stages of Polymer Crystallization by Simultaneous Small- and Wide-Angle X-ray Scattering", Macromolecules, 33(3), 978-989 (2000).
- C. Park, S. Simmons, L. J. Fetters, B. Hsiao, F. Yeh and E. L. Thomas, "Spherical to Cylindrical Microdomain Transformation by Application of a Flow Field", Polymer, 41(8), 2971-2977 (2000).
- Zhi-Gang Wang, Xuehui Wang, Benjamin S. Hsiao, Roger A. Phillips, Francisco J. Medellin-Rodriguez, Srivatsan Srinivas, Charles C. Han, "Structure and Morphology Development in Syndiotactic Polypropylene during Isothermal Crystallization and Subsequent Melting", J. Polym. Sci., Polym. Phys., 39 (23), 2982-2995 (2001).

## **C. Collaborators within the Past 48 Months - other than on the Listed Publications.**

Francisco Baltá-Calleja, Stephen Z. D. Cheng, Benjamin Chu, Julia Kornfield, Joe Lichtenhan, Sanjeeva Murthy, M. Muthukumar, Roger Phillips, Miriam Rafailovich, Rick Register, Jim Runt, Hong J. Sue, Bryan B. Sauer, Jerold M. Schultz, Richard S. Stein, Norbert Stribeck, Edwin L. Thomas, Andy Tsou, Jack Zhou

## **Current Graduate Students and Postdoctoral Fellows.**

Graduate Students: Steven X. Zong, Bruce X. Fu, Ling Yang, Meiki Yu, Jonathan Chiu

Postdoctorals and Visiting Scientists: Fengji Yeh (97-), Zhigang Wang (97-), Lizhi Liu (97-), DuFei Fang (97-), Shaofeng Ran (98-), KwanSok Kim (00-), Raj Somani (99-), Michael Gelfer (00-), Igors Sics (00-), Shigeyuki Toki (01-), Daisuke Kawakami (01-)

## **Graduate and Postdoctoral Advisors.**

Graduate Advisors: Montgomery T. Shaw and Edward T. Samulski

Postdoral Advisors: Richard S. Stein and H. Henning Winter

## **BENJAMIN CHU - RESEARCH SUPPORT (TOTAL COST)**

### ACTIVE

- |    |   |   |     |
|----|---|---|-----|
| 1. | 2 R01 HG01386-07 (Chu)<br>National Institutes of Health<br>Separation Media for DNA Capillary<br>and Micro-Chip Based Electrophoresis | 9/01/01 – 8/31/04<br>\$811,026  | 10% |
| 2. | DAAD19011-394<br>DoD ARO-DURIP (PI: Chu; Co-PI: Hsiao)<br>Micro-Processing System for Lightweight<br>Flexible Nanocomposites          | 04/01/01-03/31/02<br>\$78,850<br>(including \$23,655 in matching funds) | 5%  |
| 3. | DMR 9984102 (Chu)   | 1/1/00 – 12/31/02   | 10% |



**Unique Hybrid Multi-Jet Electrospinning and Melt-Blown Technology for Mass Production of Nanostructured Membranes**

National Science Foundation Nanostructures and Activity of Polyelectrolyte/ Surfactant Complexes	\$285,000	
4. DAAD 190010419 (Chu) U.S. Army Research Office Dynamic Studies in Fiber Processing (SUNY/Stony Brook)	7/01/00 – 6/30/03 \$347,160	10%
5. DEFG0286ER45237.016 Department of Energy (Chu) Modification of Nanostructured Materials	1/1/01 – 12/31/03 \$338,968	10%
6. DEFG0299ER45760 (Hsiao, PI; Chu, Co-PI) Department of Energy Support for the Advanced Polymers Beamline at the National Synchrotron Light Source	3/15/99 – 3/14/02 \$300,000	5%
7. CARS: A National Chemistry and M National Science Foundation (PI: J. P. Viccaro) ChemMat aterials Synchrotron Research Facility at the Advanced Photon Source (APS) (Co-PIs: B. Chu, P. Coppens, S. Rice, M. Schlossman, P. S. Pershan).	\$5,563,166 3/15/01 – 2/28/06 (includes of supplement of \$240,000 per year from DoE)	5%
8. National Science Foundation (PI: M. Rafailovich) MRSEC for Polymers at Engineered Interfaces (Co-PIs: B. Chu, J. Sokolov, B. Hsiao, D. Gersappe);	6/01/01 – 5/31/06 \$4,093,283	5%
9. NIH-SBIR (PI: Dufei Fang; Co-PIs: B. Chu/B.Hsiao) Bioabsorbable Nanostructured Membranes for Prevention of Post-Operative Adhesions	05/01/01-04/30/03 \$399,294	5%
10. Center for Biotechnology (Co-PI: B. Chu) Fabrication of Bioabsorbable Membranes For Prevention of Post-Operative Adhesions	07/01/01-06/15/02 \$35,000	5%

**RESEARCH SUPPORT (TOTAL COST)**

**PI: BENJAMIN S. HSIAO**

**ACTIVE**

1. Clemson University Research Foundation (Hsiao) (as subcontractor on their NSF grant) In-Situ X-Ray Studies of Polymer Fiber/Film Deformation	5/1/00 – 9/30/02 \$122,500	5%
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**Unique Hybrid Multi-Jet Electrospinning and Melt-Blown Technology for Mass Production of Nanostructured Membranes**

2.	Center for Biotechnology (Co-PI: B. Chu) Fabrication of Bioabsorbable Membranes For Prevention of Post-Operative Adhesions	07/01/01-06/15/02 \$35,000	5%
3.	DEFG0299ER45760 (Hsiao, PI; Chu, Co-PI) Department of Energy Support for the Advanced Polymers Beamline at the National Synchrotron Light Source	3/15/99 – 3/14/02 \$300,000	10%
4.	ExxonMobil Chemical Company (Hsiao) Flow Induced Crystallization in Polymers	2/15/00 – 2/14/03 \$181,000	10%
5.	National Institute of Standards & Technology (Hsiao) Characterization and modeling of Phase Separation and Crystallization of Polyethylene Blends	09/16/00-09/15/03 \$187,157	5%
6.	National Science Foundation (PI: M. Rafailovich) MRSEC for Polymers at Engineered Interfaces (Co-PIs: B. Chu, J. Sokolov, B. Hsiao, D. Gersappe);	6/01/01 – 5/31/06 \$4,093,283	5%
7.	National Science Foundation (Hsiao) Orientation-Induced Crystallization in Polymers	04/01/01-03/31/04 \$328,577	10%
8.	DoD ARO-DURIP (PI: Chu; Co-PI: Hsiao) Micro-Processing System for Lightweight Flexible Nanocomposites	04/01/01-03/31/02 \$78,850 (including \$23,655 in matching funds)	5%
9.	NIH-SBIR (PI: Dufei Fang; Co-PIs: B. Chu/B.Hsiao) <b>Bioabsorbable Nanostructured Membranes for</b>  Prevention of Post-Operative Adhesions	05/01/01-04/30/03 <b>\$399,294</b>	5%

**Small Business Innovation Research (SBIR) Program  
Cost Proposal**

Firm: Stonybrook Technology and Applied Research, Inc.  
Address: P.O. Box 1336  
Stony Brook, NY 11790

Location Where Work Will Be Performed: Rm. 416, Chemistry Bldg., SUNY/SB, Stony Brook, NY 11794-3400

Proposal #: A022-2903

Title of Proposed Effort: Novel Clothing Nonwoven Liner Material - Nanofibers in Melt Blown Media

Firm's Taxpayer ID: 11-3503753

CAGE Code:

DUNS: 101394430

Topic Number: A02-193

Topic Title: Novel Clothing Nonwoven Liner Material - Nanofibers in Melt Blown Media

**TOTAL DOLLAR AMOUNT FOR THIS PROPOSAL:**

**\$119,864.40**

**DIRECT LABOR:**

	Phase I:			Option:		
Category and/or Individual:	Rate/Hour	Est. Hours	Cost	Rate/Hour	Est. Hours	Cost
Dufei Fang	31.25	200	6,250.00	31.25	250	7,812.50
Post-Doc Researcher	16.50	1040	17,160.00	16.50	680	11,220.00
<b>Subtotal Direct Labor (DL):</b>			<b>23,410.00</b>			<b>19,032.50</b>
Fringe Benefits, if not included in Overhead, (rate 30.6000 %) x DL =			7,163.46			5,823.95
Labor Overhead (rate 15.0000 %) x (DL + Fringe) =			4,586.02			3,728.47
<b>Total Direct Labor (TDL):</b>			<b>35,159.48</b>			<b>28,584.92</b>

**DIRECT MATERIAL COSTS:**

	Phase I:	Option:
Melt-blown Assembly	20,000.00	7,500.00
Multiple-jet Electrospinning Assembly*	8,000.00	2,800.00
Lab supplies & testing materials	2,200.00	2,000.00
<b>Subtotal Direct Materials Costs (DM):</b>	<b>30,200.00</b>	<b>12,300.00</b>
Material Overhead (rate 15.0000 %) x DM:	4,530.00	1,845.00
<b>Total Direct Materials Costs (TDM):</b>	<b>34,730.00</b>	<b>14,145.00</b>

**OTHER DIRECT COSTS:**

	Phase I:	Option:
Lab rental, utilities	0.00	4,300.00
Shop services	0.00	2,000.00
<b>Subtotal Other Direct Costs (ODC):</b>	<b>0.00</b>	<b>6,300.00</b>
Direct Cost Overhead (rate 15.0000 %) x ODC	0.00	945.00
<b>Total Other Direct Costs (TODC):</b>	<b>0.00</b>	<b>7,245.00</b>

G&A (rate 0.0000 %) x (base: TDL)

0.00

0.00

Total Cost:

69,889.48

49,974.92

Fee or Profit (rate 0.0000 %)

0.00

0.00

**TOTAL ESTIMATED COST:**

**69,889.48**

**49,974.92**

**Explanatory material relating to the cost proposal:**

1. We have to build a new melt-blown assembly, estimated initial investment is about \$25,000. We request \$20,000 and another \$7,500 in the optional period. 2. The cost of multiple jet is much higher than we requested. STAR is going to subsidise the investment for the assmebly. 3. Testing materials include polymers, solvent etc.

*The cost breakdown portion must be signed by a responsible official.*

Sign: Dufei Fang Date: 8/14/02

Name: Dufei Fang Title: Director of Technology

>>Has any executive agency of the United States Government performed any review of your accounts or records in connection with any other government prime contract or subcontract within the past twelve months? No

>>Will you require the use of any government property in the performance of this proposal? No

>>Specify the type of payment desired: Partial payments

Subj: **Army 02.2 Phase I SBIR Solicitation**  
Date: 11/11/2002 3:51:14 PM Eastern Standard Time  
From: MeagherK-Contractor@hqamc-exchg.army.mil  
To: dfangstar@aol.com  
Sent from the Internet (Details)



REPLY TO  
THE ATTENTION OF

**DEPARTMENT OF THE ARMY**  
**UNITED STATES ARMY RESEARCH LABORATORY**  
**UNITED STATES ARMY RESEARCH OFFICE - WASHINGTON**  
**5001 EISENHOWER AVENUE (ROOM 8N31)**  
**ALEXANDRIA, VIRGINIA 22333-0001**

November 11, 2002

Dr. Dufei Fang  
Stonybrook Technology and Applied Research, Inc.  
P.O. Box 1336  
Stony Brook, NY 11790

Subject: Army 02.2 Phase I SBIR Solicitation  
Topic #: A02-193  
Control #: A022-2903

Dear Dr. Fang:

This letter informs you that your proposal entitled "Novel Clothing Nonwoven Liner Material - Nanofibers in Melt Blown Media" submitted to the Army Small Business Innovation Research (SBIR) Program, has been competitively selected for negotiation and possible contract award. The contract award to fund your proposed work is contingent upon successful negotiations through the assigned Contracting Officer and availability of funds. Consequently, please be advised that the Government is not responsible for any funds expended by offerors prior to award of signed contracts.

You will soon be contacted by a designated procurement official within the cognizant Army Laboratory or Center to initiate the contract award process.

Sincerely,

Janice M. Baker  
MAJ, USA  
Army SBIR Program Manager

Tuesday, November 12, 2002 America Online: DFang STAR

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